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Effect of Mechanical Alloying Atmospheres and Oxygen Concentration on Mechanical Properties of ODS Ferritic Steels

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1. Introduction

Oxide dispersion strengthened (ODS) steel is the most promising candidate for a core structural material for next-generation nuclear systems such as a Gen. IV fission and DEMO fusion reactor. This is due to its excellent elevated temperature strength and irradiation resistance. Finely dispersed nano-oxide particles with a high number density in the homogeneous grain matrix are essential to achieve superior mechanical properties at high temperatures, and these unique microstructures can be obtained through the mechanical alloying (MA) and hot consolidation process. The microstructure and mechanical property of ODS steel significantly depends on its powder property and the purity after the MA process. These contents should be carefully controlled to improve the mechanical property at elevated temperature. In particular, appropriate the control of oxygen concentration improves the mechanical property of ODS steel at high temperature [1]. An effective method is to control the mechanical alloying atmosphere by high purity inert gas.

In the present study, the effects of mechanical alloying atmospheres and oxygen concentration on the mechanical property of ODS steel were investigated.

2. Methods and Results

2.1 Experimental procedure

The ODS alloys used in this study are Fe-15Cr-1Mo-0.3Ti with Y_2O_3 in wt%. The ODS alloys were fabricated by mechanical alloying and a hot isostactic pressing (HIP) process. Metallic raw powders and Y_2O_3 powder were mechanically alloyed by a horizontal ball-mill apparatus, Simoloyer CM-20. Mechanical alloying atmospheres are thoroughly controlled in ultra-high purity argon (99.9999%), a mixture gas (Argon-4vol.% Hydrogen, 99.999%), and helium (99.999%). To reduce an excess oxygen concentration, YH_2 was added instead of Y_2O_3 according to the stoichiometry ratio. The mechanical alloying was performed at an impeller rotation speed of 240 rpm for 48h with a ball-to-powder weight ratio (BPWR) of 15:1. After the mechanical alloying, the particle distribution was measured by a laser diffraction scattering method using a particle size analyzer. SEM was utilized to observe the surface morphology of MA powders. The chemical composition and oxygen concentration of MA powders were analyzed by an ICP-AES and KS D 1778 methods, respectively. MA powders were then sieved and charged in a stainless steel capsule. All powder handling processes for the weighing, collecting, sieving, and charging were conducted in a completely controlled high purity argon atmosphere to prevent oxygen contamination during the process. Sealed capsules were then degassed at 400°C below 5×10^{-4} torr for 3h. The HIP was carried out at 1150°C for 3h at a heating rate of 5°C/min and the following furnace cooling. Hot rolling at 1150°C was done in a fixed rolling direction for the plate shape with 65% of the total reduction rate. Detailed conditions for mechanical alloying process are summarized in Table 1. The grain morphology was observed by FE-SEM. Specimens for mechanical property evaluations were taken out in the rolling direction. Creep rupture test were carried out at 700°C.

2.2 Powder property and its oxygen concentration after MA processes

The surface morphologies of MA powder after MA processes in various atmospheres are shown in Fig. 1. Powders milled in the Ar were irregularly spherical and flake shapes with a somewhat rough surface. A more spherical shape with a smooth surface was observed in a mixture gas and He milled MA powders. The spherical shape normally gives an advantage for a higher charging density and the smooth surface contributes to a lower oxygen contamination owing to less adhesion sites of oxygen on the surface. The mean particle size of Ar milled MA powder is smaller than the mixture gas. MA powder in He has the largest mean particle size among three kinds of MA atmospheres. Meanwhile, prior particle boundary (PPB) pores usually affect the creep property of ODS steels, because they become the origin of the fracture and develop into large creep cavities when deformed at high temperature. However, H. Sakasegawa et al. reported that the size of MA powder does not affect the creep cavities or microstructural features [2].

Table I: MA process conditions of ODS ferritic alloys

	Milling time (hrs)	Milling speed (rpm)	BP WR	Atm.	Remarks
1	48	240	15:1	Ar	Standard
2	48	240	15:1	Ar-H_2	H_2 atm.
3	48	240	15:1	He	He atm.
4	48	240	15:1	Ar	YH_2 addition

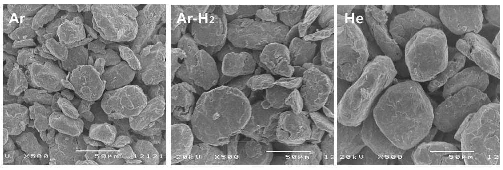

Fig. 1. Surface morphologies of alloy powders after MA processes in various atmospheres

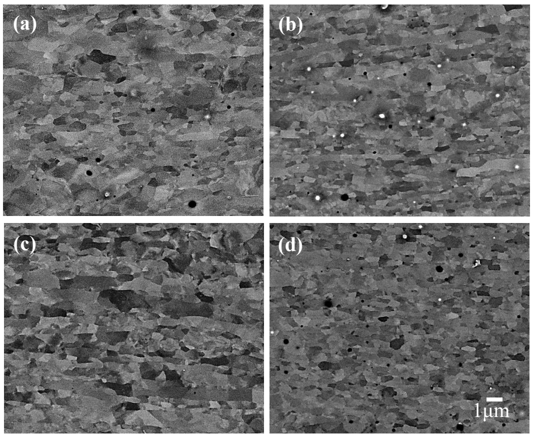

Fig. 2. Grain morphology of ODS ferritic alloys milled in different atmospheres and oxygen concentrations; (a) Ar, (b)Ar-H_2, (c) He, and (d) YH_2 substitution.

2.3 Microstructures and mechanical properties of ODS ferritic alloys

The SEM images of the grain morphology observation of ODS ferritic alloys milled in different atmospheres and oxygen concentrations are shown in Fig. 2. Grains are elongated toward the hot rolling direction which is parallel to horizontal direction in the images. It seems to incur secondary recrystallization during hot rolling process at 1150°C because elongated grains are clearly distributed. The grain distribution of ODS alloy milled in the mixture gas and He is quite homogeneous, while Ar milled ODS alloy shows the co-existence of fine (<1μm) and coarse (>5μm) grains. However, YH_2 substituted ODS alloy to reduce the oxygen concentration, shows a very fine and uniform grain distribution. The microstructural inhomogeneity of Ar milled ODS alloy is attributed to a high oxygen concentration in ODS alloys. The excess oxygen of Ar milled ODS alloy is higher than those of mixture gas, as shown in Table 2.

Creep rupture tests of ODS ferritic alloys with different excess oxygen concentrations were performed at 700°C under various loads between 100 and 170MPa. The test results are plotted at the log-log scale in Fig. 3. The creep strength of YH_2 added ODS alloy is more superior than other alloys. This is ascribed to the excess oxygen in ODS ferritic alloy. YH_2 added ODS alloy has the least excess oxygen, owing to an oxygen-reduction reaction between YH_2 and contaminated oxygen from raw materials or the penetration during the MA process.

Table II: Chemical compositions of ODS ferritic alloy

	Fe	Cr	Mo	Ti	Y	O	Y_2O_3	*Ex.O
Ar	bal.	14.84	0.98	0.30	0.26	0.30	0.33	0.23
Ar-H_2		14.85	0.99	0.31	0.26	0.25	0.33	0.18
He		14.81	0.99	0.31	0.26	0.23	0.33	0.16
YH_2		15.03	1.00	0.30	0.26	0.18	0.33	0.11

*Ex.O = Total oxygen conc. - oxygen conc. in Y_2O_3

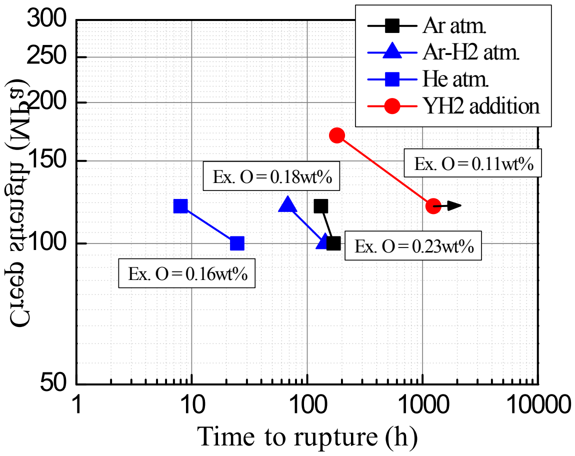

Fig. 3. Creep strength of ODS ferritic alloys milled in various atmospheres and excess oxygen

3. Conclusions

ODS ferritic alloys were fabricated in various atmospheres, and the HIP process was used to investigate the effects of MA atmospheres and oxygen concentration on the microstructure and mechanical property. ODS ferritic alloys milled in an Ar-H_2 mixture, and He is effective to reduce the excess oxygen concentration. The YH_2 addition made an extremely reduced oxygen concentration by the internal oxygen-reduction reaction and resulted in a homogeneous microstructure and superior creep strength.

Acknowledgements

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